



DAVID W. TAYLOR NAVAL SHIP RESEARCH AND DEVELOPMENT CENTER



Bethesda, Md. 20084

BURNER-RIG SIMULATION OF LOW-TEMPERATURE HOT CORROSION

by Louis F. Aprigliano

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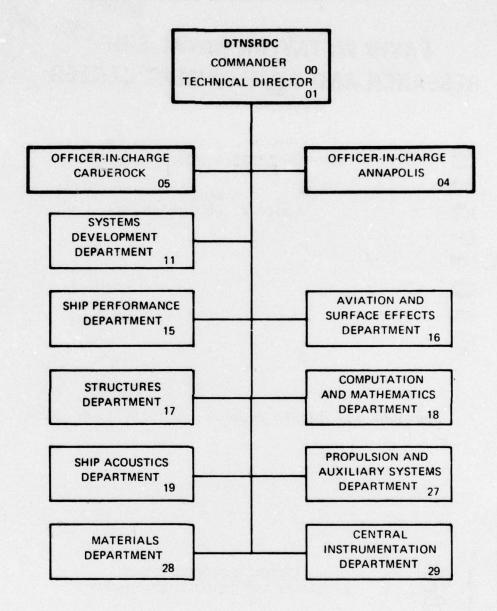
Burner-Rig Simulation of Low-Temperature Hot Correson

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20. ABSTRACT (Cont)

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(Author)



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LIST OF ABBREVIATIONS

atm - atmosphere

bal - balance

° C - degree Celsius cm - centimetre

CoCrAly - cobalt, chromium, aluminum and yttrium

° F - degree Fahrenheit

µm - micrometre mm - millimetre

mg/cm² - milligrams per square centimetre

p/m - parts per million

SEM-EDXA- scanning electron microscope - energy dispersive

X-ray analysis

wt % - weight percent

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ABSTRACT

Low-velocity burner-rig tests were conducted at four temperatures: 1200° F* (649° C), 1300° F (704° C), 1400° F (760° C) and 1550° F (844° C) in an attempt to simulate the low-temperature (circa 1300° F (704° C)) attack that is occurring on marine gas turbine hot section components. The tests were made by exposing CoCrAly-coated Rene 80 test pins to a burner-rig environment burning marine diesel fuel containing 1 weight percent S at a 30 to 1 air-to-fuel ratio. The test environment contained 5 parts per million sea salt at 1200° F (649° C) and 10 parts per million at the other three temperatures. At 1200° F (649° C) and 1400° F (760° C) after 750 hours, there was little or no hot-corrosion attack. At 1550° F (844° C) after 750 hours, typical hightemperature, hot-corrosion attack had occurred. At 1300° F (704° C) lowtemperature, hot-corrosion attack was duplicated within 450 hours. The occurrence of low-temperature attack was found to be related to the presence of CoSO,

INTRODUCTION

Operation of main propulsion marine gas turbine engines at low or intermediate power levels results in relatively low temperatures on the blade metal. In one such engine of interest to the Navy, the LM2500, operation at low power (12,000 horsepower or less) results in blade metal temperatures of 1100° to 1350° F (593° to 732° C).¹ At these temperatures, hot-corrosion attack on the CoCrAly coated first-stage turbine blades on the LM2500 powered GTS Callaghan has been reported¹ to be occurring at rates equal to or greater than those at high-power, high-temperature operation. Since the LM2500 powered DD963 class destroyers operate at low or intermediate power levels over 80% of the time,² low-temperature, hot-corrosion attack can have a detrimental effect on engine performance.

^{*}Definitions of abbreviations used in this text are given on page i.

¹ Superscripts refer to similarly numbered entries in the Technical References at the end of the text.

The morphology of high-temperature (circa 1700° F (926°C)) hot-corrosion attack of CoCrAlY coatings is characterized by depletion of aluminum from the coating to form Al_2O_3 .¹ This causes a layer depleted of the aluminum-rich beta phase to form beneath the oxide/coating interface. As the attack progresses, a network of Al_2O_3 and depleted coating forms. The cobalt and chromium in the remaining portions of depleted coating are oxidized at the surface which completes the degradation of the coating. Sulfides do not appear in the depletion zone until the aluminum-rich phase is completely consumed.

With low temperature (circa 1300° F (704° C)) hot corrosion aluminum depletion does not occur.¹ Instead there is almost uniform conversion to oxide scales of all coating constituents at about the same rate. There is, however, some chromium depletion beneath the scale/coating interface.¹ The scale in contact with the coating is high in aluminum and chromium and low in cobalt. Frequently, a loosely adherent outer scale rich in cobalt is formed over this inner scale. Sulfur is found in the scale particularly at the scale/coating interface.¹

As part of any development of a coating that can resist low-temperature as well as high-temperature attack, a laboratory test that can duplicate this engine attack must be developed. Once this is done, it is then possible to screen promising coatings for their resistance to both temperature levels of hot-corrosion attack. Such an approach can reduce the amount of expensive engine testing needed to qualify coatings for Fleet use. Since test procedures already exist to duplicate high-temperature hot corrosion, the objective of this work was to develop a test that can simulate low-temperature hot corrosion in the laboratory.

EXPERIMENTAL PROCEDURE

MATERIALS

Pins 1/8 inch (0.32 cm) in diameter by 1 1/2 inches (3.8 cm) long of nickel-base superalloy Rene 80 were tested with a CoCrAly coating. The Rene 80 was in the equiaxed, conventionally cast form. The CoCrAly coating was applied by physical vapor deposition and had a nominal thickness of 0.005 inch (0.127 mm). The particular CoCrAly coating tested was of the BC21 composition developed by the General Electric Company. This coating/substrate combination was selected since this is the same combination used for the first-stage, high-pressure turbine blades of the LM2500 powered, DD963 class destroyers. Coated pins from two suppliers were used for these tests. Pins from supplier A were used in the 1400° F (760° C) burner-rig run described below while pins from supplier B were used in the other three burner-rig runs. The chemical compositions of Rene 80 and BC21 are shown in table 1.

Table 1 CHEMICAL COMPOSITION OF RENE 80 AND BC21

Material	Composition, wt %										
Designation	Ni	Со	Cr	Al	Y	Ti	W	Мо	C	В	Zr
Rene 80 (nominal composition)	Bal	9.5	14.0	3.0	-	5.0	4.0	4.0	0.17	0.015	0.03
BC21 (specification range)	-	Bal	20/26	11/13	0.1/0.5	-	-	-	-	-	-

BURNER-RIG TESTS

The test pins described above were exposed to the hot-corrosion environment in a low-velocity, atmospheric-pressure burner rig. The operation of the burner rig has been previously described. A diagram of the apparatus cross section is shown in figure 1. Four gas-stream temperatures were selected for these tests: 1200° F (649° C) and 1300° F (704° C) as these are temperatures within the range associated with low-temperature attack of LM2500 first-stage, high-pressure turbine blades on TS Callaghan, and 1400° F (760° C) and 1550° F (844° C) as are the temperatures which the Admiralty Materials Labora-and the Cranfield Institute of Technology in England, pectively, associate with the maximum rate of low-temperature attack. The other significant test parameters for each gas-stream temperature are listed as follows:

- 5 p/m at 1200° F (649° C) ASTM sea salt in air concentration 10 p/m at the other three temperatures - Marine diesel, 1 wt % S Fuel Air/fuel ratio - 30/1 - 950 hours at 1300° F (704° C) Total test duration 750 hours at the other three temperatures - Every 100 hours to room temper-Thermal cycling ature in still air, with 1 cycle of 50 hours

The sulfur content of the fuel is purposely raised to 1 wt % by adding tertiary butyl disulfide. Two BC21-coated pins were exposed at each temperature. One specimen was removed after 450 to 500 hours of exposure. If the attack was negligible after this time, the other BC21-coated pin was exposed for an additional 250 hours. If there was significant attack after 400 to 500 hours, testing was continued until it was estimated that the BC21 coating would be penetrated through to the Rene 80.

High-purity platinum pins, 1/8 inch (0.32 cm) diameter by 3/4 inches (1.9 cm) long, were exposed along with the BC21-coated pins to measure the amount of salt deposited during each of the four burner-rig runs. The platinum pins were weighed before and after exposure, and the weight gain was then normalized to the area exposed. These data should be considered as an approximation of the amount of salt deposited on the pin, as some can flake or spall off with thermal cycling.

Surface temperature measurements on a test pin were made at a gas-stream temperature of 1300° F $(704^{\circ}$ C) under the same conditions as for the 1300° F $(704^{\circ}$ C) burner-rig run but without salt. The test pin used was a cobalt-base superalloy, X-40, and was 1/8 inch (0.32 cm) in diameter by 1 1/2 inches (3.8 cm) long. Platinum-platinum 10% rhodium thermocouples were attached to the top and bottom of the test pin as shown in figure 2. The surface temperatures at these points were then recorded for 24 hours while the carousel and pin rotated in the burner rig.

EVALUATION

After testing, the BC21 pins from the 1300° F (704° C), 1400° F (760° C), and 1550° F (844° C) runs were washed with distilled water. The water wash from each pin was then analyzed for cobalt, sodium, and iron. Water-soluble cobalt is an indicator of the presence of CoSO4; iron is an indicator of ferric sulfate contamination or formation in the burner-rig environment; sodium is an indicator of salt deposition. The washes from several of the pins were also analyzed for chloride, sulfate, magnesium, and calcium, all of which are constituents of sea salt. The cobalt, sodium, magnesium, calcium, and iron analyses were performed by atomic absorption. The chloride and sulfate analyses were performed by standard titration techniques.

The salt deposited on the platinum pins was removed by soaking the pins in distilled water. The water was then evaporated, leaving a salt residue. This salt and the loosely adherent scale which formed on the BC21 pins from the 1300° F (704° C) run were analyzed by X-ray-diffraction techniques.

The BC21 pins were sectioned and prepared for metallography and scanning election microscopy after the distilled water washing. The specimens were mounted in a copper-bearing material to which silicon nitride was added to aid in edge retention. Finally, the specimens were ground through 600-grit silicon carbide paper and then polished through 0.25 µm diamond.

RESULTS AND DISCUSSION

1200° F (649° C) AND 1400° F (760° C) RIG RUNS

The pins exposed to the burner-rig tests at 1200° F (649° C) and 1400° F (760° C) underwent very little if any hot-corrosion attack. This is evident from the condition of the pins before and after exposure, as shown in figure 3. Significant hot-corrosion attack failed to occur even with the appreciable deposition of salt at each of the temperatures. Specifically, the amount of salt deposited on the platinum pins as listed in table 2 was 6.2 mg/cm² in 750 hours at 1200° F (649° C) and 24 mg/cm² in 750 hours at 1400° F (760° C). X-ray diffraction of the deposits on the platinum pins showed the salt to be Na₂SO₄; NaCl was not found in these deposits.

TABLE 2
AMOUNT OF SALT DEPOSITED ON PLATINUM PINS

Temperature	Amount of Salt Deposited for Time Shown						
1200° F (649° C)	0.94 mg/cm ² in 100 hours		6.2 mg/cm ² in 750 hours				
1300° F (704° C)	-	8.8 mg/cm ² in 450 hours					
1400° F (760° C)	-	14.4 mg/cm ² in 500 hours	24 mg/cm2 in 750 hours				
1550° F (844° C)	3.9 mg/cm^2 in 200 hours	7.5 mg/cm ² in 400 hours					

It should not be inferred from this result and those to be presented later that NaCl was not deposited on the platinum pins or CoCrAlY specimens. NaCl can be deposited by condensation from the vapor state or by impaction of solid salt particles. ^{6,7} However, once deposited, the NaCl has been found to survive only for very short periods of time; it can be vaporized from the surface or converted to Na₂SO₄. Shores, et al, ⁸ propose the reaction for this conversion with constituents in the combustion gas as follows:

2 NaC1 + SO₂ + O₂
$$\longrightarrow$$
 Na₂SO₄ + C1₂
2 NaC1 + SO₂ + H₂O + 1/2O₂ \longrightarrow Na₂SO₄ + 2HC1

Thus, conversion to sulfate and/or evaporation would account for the removal of NaCl from the deposit.

The 1200° F (649° C) pins were not water washed. The results of the water wash from the CoCrAly pins exposed at

1400° F (760° C) showed major amounts of Na and SO4, minor amounts of Ca and Mg, and little or no Fe, Co, and Cl (see table 3). These results agree with the X-ray determination of Na₂SO₄ as the major salt found on the platinum pins. The fact that cobalt was not found in significant quantities in the 1400° F (760° C) water wash is important and will be discussed in greater detail along with the 1300° F (704° C) burner-rig results.

TABLE 3
ANALYSIS OF WATER WASH

	Time,	P/M							
Temperature	hours	Ca	Fe	So4 ⁽¹⁾	Na	Ca	Mg	C1 ⁽¹⁾	
1300° F (704° C)	500(2) 950(2)	4.35 1.68	0.11	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	5.4 28.0	(4)	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	(5)	
1400° F (760° C)	500(3) 7 50(3)	0.19 0.12	0.00	58 11 9	2 7. 5 44.8	(4) 2.5	(4)	1.2	
1550° F (844° C)	500(3) 750(3)	0.00	0.00	46 1 84	14.1 66.8	0.1	0.0	0	
Detection 1	0.01	0.01	1	0.2	0.1	0.1	1		

(1) Analysis of SO₄ and C1 by standard titration techniques, all others by atomic absorption.

- (2) Original sample size 100 ml.
- (3) Original sample size 250 ml.
- (4) Not analyzed for.
- (5) Chloride analysis contaminated.

1550° F (844° C) RIG RUN

Macro and Micro Examination

The pins exposed to the 1500° F (844° C) burner-rig test underwent hot-corrosion attack. The attack was localized in nature, and after 750 hours the maximum depth of attack was two mils. The condition of the pins before and after testing is shown in figure 4. The hot-corrosion attack shown in item (b) of figure 4 is not characteristic of that associated with low-temperature attack but is typical of high-temperature hot corrosion. There is a layer within the coating beneath the oxide scale that is characterized by the absence of the aluminum-rich beta phase (grey phase) and which appears as a white layer on the photomicrograph (item (b) of figure 4). SEM-EDAX analysis (item (b) of figure 5) has shown this layer to be depleted of aluminum and rich in cobalt and chromium. The aluminum depletion is the result of the formation of an Al203 oxide scale.

The oxide fingers extending into the depletion layer itself are also rich in aluminum (item (c) of figure 5). Sulfide particles were found in advance of these oxide fingers, and SEM-EDAX analysis (items (d) and (e) of figure 5) showed these particles to be rich in sulfur and chromium with detectable amounts of aluminum and cobalt.

It should be noted that the CoCrAly coating on the pins used in this burner-rig run (and the 1300° F $(704^{\circ}$ C) run) had, in the untested condition, a broad band of aluminum-rich beta phase located 0.001 to 0.002 inch (0.025 to 0.050 mm) from the coating/superalloy interface (item (a) of figure 4). After 750 hours at 1550° F $(844^{\circ}$ C) the microstructure of the coating was far more uniform with only a thin band of the beta phase still present (item (b) of figure 4). The hot-corrosion attack did not penetrate to this band during the test, and it is therefore not known whether this beta band would have accelerated or impeded the attack under the conditions of this 1550° F $(844^{\circ}$ C) burnerig run.

Deposit Analysis

X-ray diffraction of the deposit on the platinum pins revealed Na_2SO_4 . Once again NaCl was not found in the deposit. The amount of salt deposited was 7.5 mg/cm² in 400 hours (see table 2).

The results of the water wash from the CoCrAly pins exposed at 1550° F (844° C) showed significant Na and SO₄ and little or no Co, Fe, Ca, Mg, or Cl. These results are similar to those for the 1400° F (760° C) run and agree with the X-ray determination of Na₂SO₄ as the major salt found on the platinum pins. Once again cobalt was not found in significant quantities in the water wash, and this result will be discussed in greater detail along with the 1300° F (704° C) burner-rig run results.

1300° F (704° C) RIG RUN

Macro Examination

The condition of the pins while still mounted in the carousel holder is shown in figure 6. A dark, loosely adherent oxide scale had formed at approximately 0.3 to 0.4 inch (0.7 to 1.0 cm) from the top of the pins. This loosely adherent scale was identified as Co₃O₄ by X-ray diffraction. There was a sharply defined zone delineating this scale from the top-third of the pins, which to the unaided eye, appeared to be relatively unattacked and had a spotty surface coloring of greenish blue. The substance giving this surface coloration was so thin and adherent that it could not be removed for conventional X-ray diffraction analyses. However, it can be said that it was not CoSO₁ since CoSO₁ is water soluble and this substance was not.

Micro Examination

Metallographic examination and SEM-EDXA analysis of the pins from the 1300° F $(704^{\circ}$ C) burner-rig run revealed hot-corrosion attack that was typical of low-temperature engine attack. The degree of attack was found to change drastically along the length of the pins. In comparison to the microstructure of cross sections within the lower two-thirds of these pins (figures 8 and 9) the top-third of the pins underwent much less hot corrosion than the bottom two-thirds. In fact, the attack within the bottom two-thirds of the pins was quite advanced, with a depth of four mils after 450 hours (figure 8) and with almost complete penetration of the coating after 950 hours (item (b) of figure 9).

The morphology of the attack on the lower two-thirds of the pins (figures 8 and 9) was typical of low-temperature, hot-corrosion attack. Specifically, there is a thick scale in contact with both phases in the CoCrAly coating. The aluminum-rich beta phase (grey phase) has not been depleted in advance of the attack front. At high magnifications (900X) it can be seen that the attack front is slightly advanced in the beta phase with the gamma phase quickly consumed thereafter (item (a) of figure 10). SEM-EDAX maps revealed sulfur concentrated along this advanced attack front (item (b) of figure 10). The scale in contact with the coating is rich in Cr and Al and low in Co (items (c), (d), and (e) of figure 10). Other scans, not shown in figure 10, revealed Co concentrated in the outer oxide scale which, as previously mentioned, was identified as Co₃O₄. Within the coating itself there is some evidence of chromium depletion (item (c) of figure 10) but none whatsoever of aluminum depletion (item (d) of figure 10), the latter being the chief feature of high-temperature, hot-corrosion attack.

A broad band of beta phase is present in these coatings tested at 1300° F (704° C), as shown in figure 7. This band located 0.001 to 0.002 inch (0.25 to 0.050 mm) from the coating/ superalloy interface was also observed in the coatings tested at 1550° F (844° C), as shown in figure 4. The pins used in both these tests came from the same coating run or batch. Unlike the coatings tested at 1550° F (844° C), the microstructure of the coatings tested at 1300° F (704° C) did not become more uniform or homogeneous even after 950 hours. During the exposure at 1300° F (704° C), the hot corrosion attack did penetrate to this part of the coating on both pins. It is noteworthy that the attack has a nodular shape until it reached the beta band (figures 8 and 9). At this point the attack appears to proceed faster laterally than into the coating. It is possible that the beta band has slowed the attack. However, the beta band did not completely halt the attack as the coating was nearly penetrated in 950 hours (item (b) of figure 9).

Deposit Analysis

As was found at the other three test temperatures, the salt on the platinum pins after exposure was Na2SO4 without evidence of NaCl. As previously mentioned, it should not be inferred from these results that NaCl was not deposited on the pins. The amount of salt deposited on the platinum pins was 8.8 mg/cm2 in 450 hours (see table 2). The results of the water wash from the CoCrAly pins showed significant Na and Co with little or no Cl or Fe. The presence of Co in the water wash is important as it is representative of the presence of water soluble CoSO4 on the surface of the pins, the formation of which has been theorized as the key to low-temperature attack. This observation is consistent with the lack of low-temperature attack at 1400° F (760° C) and 1550° F (844° C) where appreciable Co was not found in the water wash. The 1200° F (649° C) pins were not water washed so a relationship cannot be drawn between the lack of attack at 1200° F (649° C) and the presence or absence of Co in the water wash.

Temperature Distribution

Due to the sharply defined boundary separating regions of unattacked and attacked coating along the length of the pins, tests were made to determine the temperature distribution along the length of the pins. Such a temperature distribution, if found to encompass the decomposition temperature of CoSO_L, might explain the sharply defined regions. The surface temperatures on an X-40 dummy pin from top to bottom were determined at the most forward or hottest point in the burner rig with a gas-stream temperature of 1300° F (704° C) to be 1320° to 1297° F (715° to 703° C) after 6 hours and 1314° to 1291° F (712° to 699° C) after 24 hours at temperature. At the coldest point, 180 rational degrees from the hottest point, the temperature distribution from top to bottom was 1297° to 1284° F (703° to 696° C) after 6 hours and 1291° to 1272° F (699° to 689° C) after 24 hours at temperature. These temperature distributions point to CoSO, decomposition as an explanation for the attack pattern. Although the CoSO4 decomposition temperature is not precisely known under the dynamic gas environment in this burner rig, Ostroff and Sanderson report the decomposition temperature as 1306° F (708° C) for a tube furnace air environment with flowing nitrogen, while Wortman et al1 report the decomposition temperature as approximately 1320° F (716° C) for a gas turbine environment of 10 atm burning a diesel fuel with 0.7 wt % S. Thus, it is likely that the temperature distribution on the pins bracketed the CoSOL decomposition temperature. If this is the case, then on surfaces with temperatures above the decomposition temperature (but too low for typical high-temperature hot corrosion), there should be little hot-corrosion attack, while on surfaces with temperatures at or near the decomposition temperature there should be noticeable hot corrosion. Indeed there was very little hot-corrosion

attack on the top-third of the pins, while on the bottom twothirds of the pins there was noticeable low-temperature, hot-corrosion attack.

SUMMARY

Low-temperature, hot-corrosion attack occurring in marine gas turbines has been simulated by using a low-velocity, atmospheric-pressure burner rig. These results were achieved by exposing CoCrAlY coated Rene 80 to a gas-stream temperature of 1300° F (704° C). A 1 wt % S marine diesel fuel was burned with an air-to-fuel ratio of 30 to 1 while 10 p/m sea salt was injected into the burner rig. There was little or no attack during similiar tests at 1200° F (649° C) and 1400° F (760° C). Typical high-temperature, hot-corrosion attack occurred at 1550° F (844° C). The occurrence of low-temperature, hot-corrosion attack was found to be related to the presence of CoSO4.

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- 9 Ostroff, A.G., and Sanderson, R.T., "Thermal Stability of Some Metal Sulphates," <u>J. Nucl. Chem.</u>, Vol. 9, pp. 45-50 (1959)

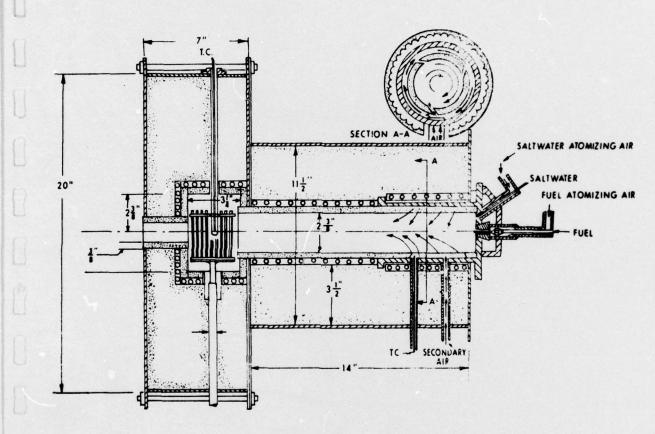


Figure 1 Cross Section of Low-Velocity, Atmospheric-Pressure Burner Rig

1 - Thermocouple Attachment 2 - X-40 Pin

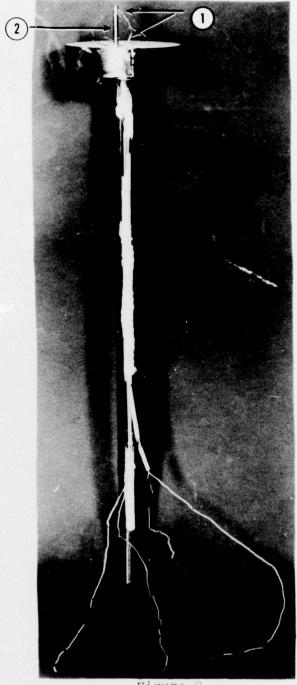
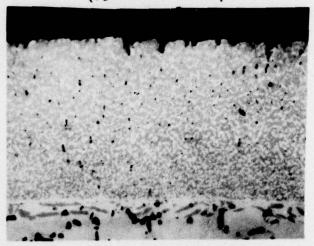
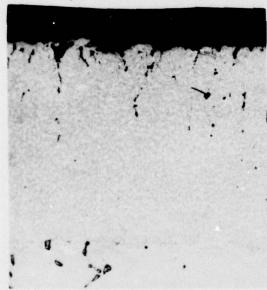


Figure 2
Thermocouple Location for
Surface Temperature Measurements

Item (a) - As-Coated
 (350X, Unetched)



Item (b) - 1200° F (649° C) (500X, Unetched)



Item (c) - 1400° F (760° C) (500X, Unetched)

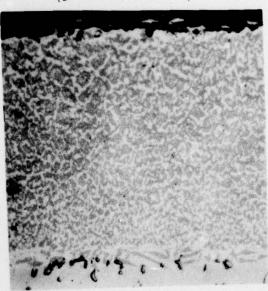
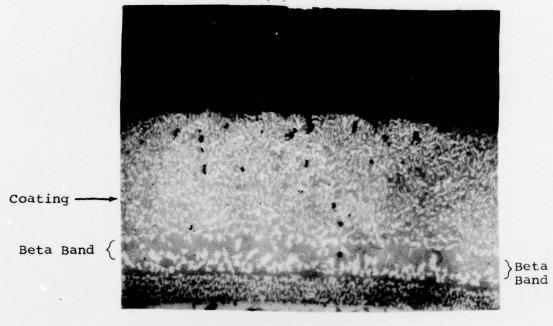


Figure 3

BC21 Coated Rene 80 Test Pins As-Coated and After Exposure at 1200° F (649° C) and 1400° F (760° C) for 750 Hours





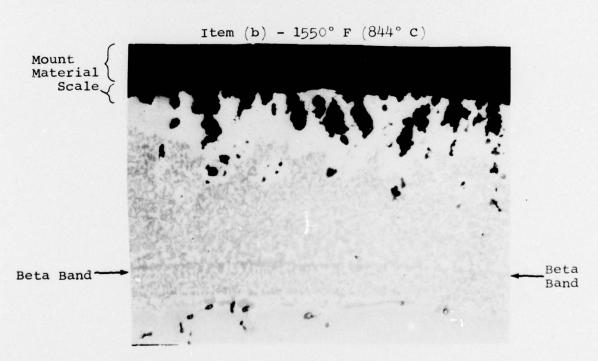
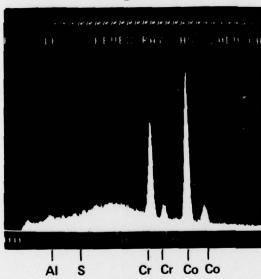


Figure 4
BC21 Coated Rene 80 Test Pins As-Coated and After Exposure at 1550° F (844° C) for 750 hours (500X, Unetched)

Item (a) - Secondary Election
Image of item (b) of Figure 4
(500X)





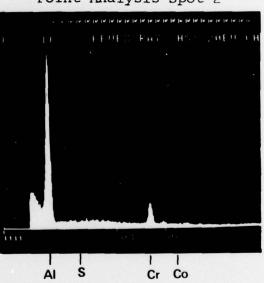
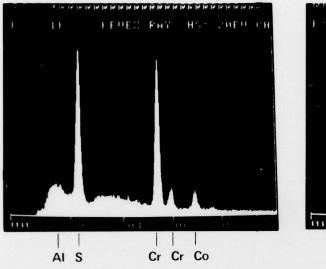


Figure 5
SEM-EDXA Analysis of BC21 Coated Rene 80
Test Pin After 750 Hours at 1550° F (844° C)

Item (d)
Point Analysis Spot 3

(d) Item (e) is Spot 3 Point Analysis Spot 4



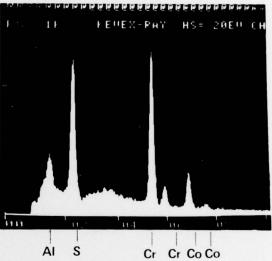


Figure 5 (Cont)

1 - Co₃0₄ Oxide Scale 2 - Platinum

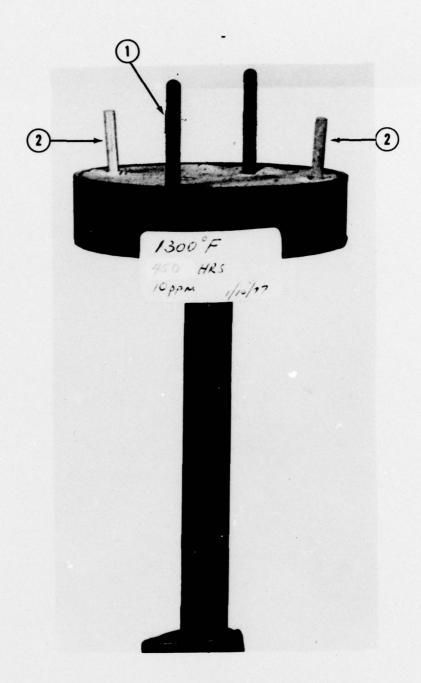


Figure 6 - Platinum Pins and BC21-Coated Rene 80 Pins After 450 Hours at 1300° F (704° C) (1X)

Mount Material {

Beta Band {

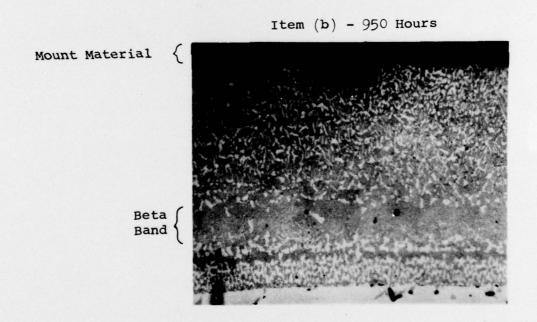


Figure 7
Microstructures Within the Top-Third of BC21 Coated Rene 80 Test Pins Exposed at 1300° F (704° C)
(500X, Unetched)

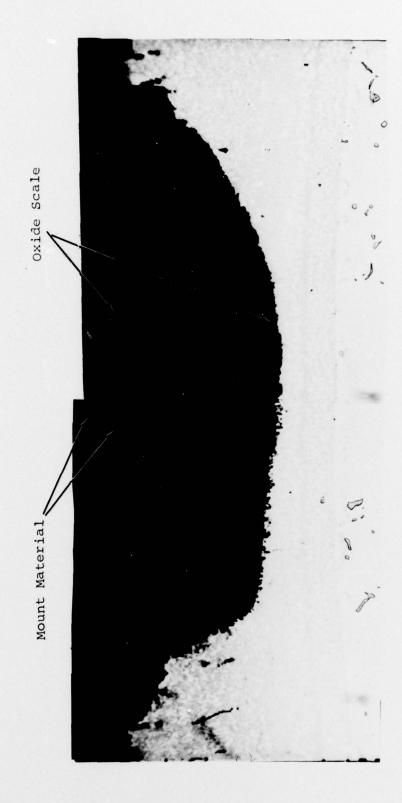
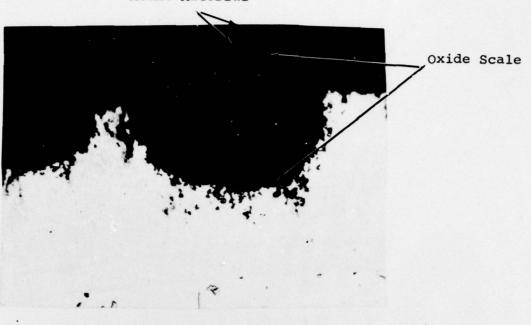


Figure 8 - Microstructure Within the Bottom Two-Thirds of the BC21 Coated Rene 80 Test Pin Exposed at 1300° F (704° C) for 450 Hours (500X, Unetched)

Item (a)

Mount Material



Item (b)

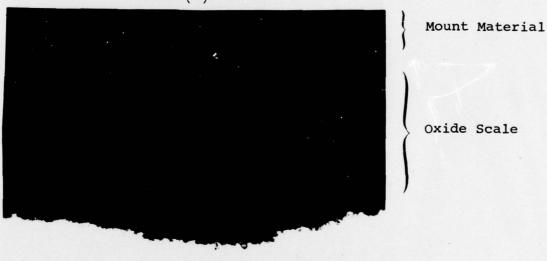


Figure 9 - Microstructures Within the Bottom Two-Thirds of the BC21 Coated Rene 80 Test Pin Exposed at 1300° F (704° C) for 950 Hours (500X, Unetched)



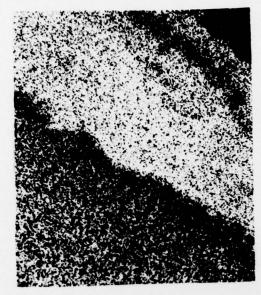
Item (a) - Back Scattered
 Electron Image (900X)

Oxide/Coating Interface

Item (b) Sulfur X-Ray Scan



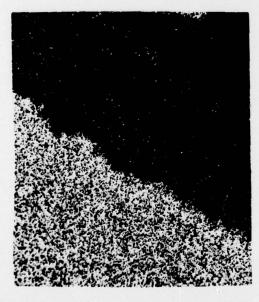
Figure 10 - SEM-EDXA Analysis of BC21 Coated Rene 80 Test Pin Exposed at 1300° F (704° C) for 450 Hours



item (c) - Chromium X-Ray Scan



Item (d) - Aluminum X-Ray Scan



Item (e) - Cobalt X-Ray Scan

Figure 10 (Cont)

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*Addressee.

MAT-77-68, November 1977